

Connecting via Winsock to STN

Welcome to STN International! Enter x:x

LOGINID:ssptanscl625

PASSWORD:

TERMINAL (ENTER 1, 2, 3, OR ?):2

\* \* \* \* \* Welcome to STN International \* \* \* \* \*

NEWS	1		Web Page for STN Seminar Schedule - N. America
NEWS	2	MAY 01	New CAS web site launched
NEWS	3	MAY 08	CA/CAPplus Indian patent publication number format defined
NEWS	4	MAY 14	RDISCLOSURE on STN Easy enhanced with new search and display fields
NEWS	5	MAY 21	BIOSIS reloaded and enhanced with archival data
NEWS	6	MAY 21	TOXCENTER enhanced with BIOSIS reload
NEWS	7	MAY 21	CA/CAPplus enhanced with additional kind codes for German patents
NEWS	8	MAY 22	CA/CAPplus enhanced with IPC reclassification in Japanese patents
NEWS	9	JUN 27	CA/CAPplus enhanced with pre-1967 CAS Registry Numbers
NEWS	10	JUN 29	STN Viewer now available
NEWS	11	JUN 29	STN Express, Version 8.2, now available
NEWS	12	JUL 02	LEMBASE coverage updated
NEWS	13	JUL 02	LMEDLINE coverage updated
NEWS	14	JUL 02	SCISEARCH enhanced with complete author names
NEWS	15	JUL 02	CHEMCATS accession numbers revised
NEWS	16	JUL 02	CA/CAPplus enhanced with utility model patents from China
NEWS	17	JUL 16	CAPplus enhanced with French and German abstracts
NEWS	18	JUL 18	CA/CAPplus patent coverage enhanced
NEWS	19	JUL 26	USPATFULL/USPAT2 enhanced with IPC reclassification
NEWS	20	JUL 30	USGENE now available on STN
NEWS	21	AUG 06	CAS REGISTRY enhanced with new experimental property tags
NEWS	22	AUG 06	BEILSTEIN updated with new compounds
NEWS	23	AUG 06	FSTA enhanced with new thesaurus edition
NEWS	24	AUG 13	CA/CAPplus enhanced with additional kind codes for granted patents

NEWS EXPRESS 29 JUNE 2007: CURRENT WINDOWS VERSION IS V8.2,  
CURRENT MACINTOSH VERSION IS V6.0c(ENG) AND V6.0Jc(JP),  
AND CURRENT DISCOVER FILE IS DATED 05 JULY 2007.

NEWS HOURS	STN Operating Hours Plus Help Desk Availability
NEWS LOGIN	Welcome Banner and News Items
NEWS IPC8	For general information regarding STN implementation of IPC 8

Enter NEWS followed by the item number or name to see news on that specific topic.

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of commercial gateways or other similar uses is prohibited and may result in loss of user privileges and other penalties.

\*\*\*\*\* STN Columbus \*\*\*\*\*

FILE 'HOME' ENTERED AT 20:46:24 ON 17 AUG 2007

=> fil reg

COST IN U.S. DOLLARS

SINCE FILE

TOTAL

ENTRY

SESSION

FULL ESTIMATED COST

0.21

0.21

FILE 'REGISTRY' ENTERED AT 20:46:33 ON 17 AUG 2007

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Property values tagged with IC are from the ZIC/VINITI data file provided by InfoChem.

STRUCTURE FILE UPDATES: 16 AUG 2007 HIGHEST RN 944884-94-0

DICTIONARY FILE UPDATES: 16 AUG 2007 HIGHEST RN 944884-94-0

New CAS Information Use Policies, enter HELP USAGETERMS for details.

TSCA INFORMATION NOW CURRENT THROUGH June 29, 2007

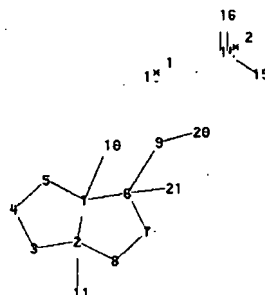
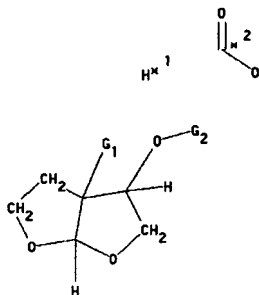
Please note that search-term pricing does apply when conducting SmartSELECT searches.

REGISTRY includes numerically searchable data for experimental and predicted properties as well as tags indicating availability of experimental property data in the original document. For information on property searching in REGISTRY, refer to:

<http://www.cas.org/support/stngen/stndoc/properties.html>

=>

Uploading C:\Program Files\Stnexp\Queries\10517966A.str



chain nodes :

9 10 11 13 14 15 16 20 21

ring nodes :

1 2 3 4 5 6 7 8

chain bonds :

1-10 2-11 6-9 6-21 9-20 14-16 14-15

ring bonds :

1-2 1-5 1-6 2-3 2-8 3-4 4-5 6-7 7-8

exact/norm bonds :

1-2 1-5 1-6 1-10 2-3 2-8 3-4 4-5 6-7 6-9 7-8 9-20 14-16 14-15

exact bonds :

2-11 6-21

G1:H,X

G2:[\*1],[\*2]

Match level :

1:Atom 2:Atom 3:Atom 4:Atom 5:Atom 6:Atom 7:Atom 8:Atom 9:CLASS 10:CLASS

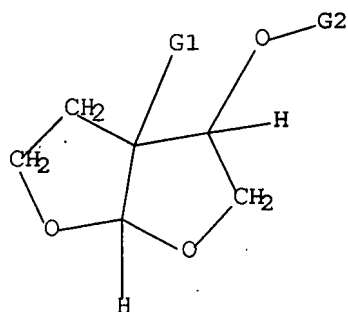
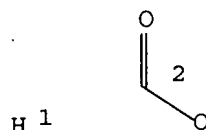
11:CLASS 13:CLASS 14:CLASS 15:CLASS 16:CLASS 20:CLASS 21:CLASS

L1 STRUCTURE UPLOADED

=> d l1

L1 HAS NO ANSWERS

L1 STR



G1 H,X

G2 [\*1],[\*2]

Structure attributes must be viewed using STN Express query preparation.

=> s sss sam l1

SAMPLE SEARCH INITIATED 20:46:57 FILE 'REGISTRY'

SAMPLE SCREEN SEARCH COMPLETED - 3917 TO ITERATE

51.1% PROCESSED 2000 ITERATIONS

0 ANSWERS

INCOMPLETE SEARCH (SYSTEM LIMIT EXCEEDED)

SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS: ONLINE \*\*COMPLETE\*\*  
 BATCH \*\*COMPLETE\*\*  
 PROJECTED ITERATIONS: 74587 TO 82093  
 PROJECTED ANSWERS: 0 TO 0

L2 0 SEA SSS SAM L1

=> s sss full l1

FULL SEARCH INITIATED 20:48:42 FILE 'REGISTRY'  
 FULL SCREEN SEARCH COMPLETED - 78321 TO ITERATE

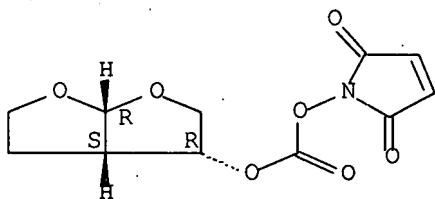
100.0% PROCESSED 78321 ITERATIONS 28 ANSWERS  
 SEARCH TIME: 00.00.01

L3 28 SEA SSS FUL L1

=> d scan

L3 28 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN  
 IN 1H-Pyrrole-2,5-dione, 1-[[[(3R,3aS,6aR)-hexahydrofuro[2,3-b]furan-3-yl]oxy]carbonyl]oxy] - (9CI)  
 MF C11 H11 N O7

Absolute stereochemistry.

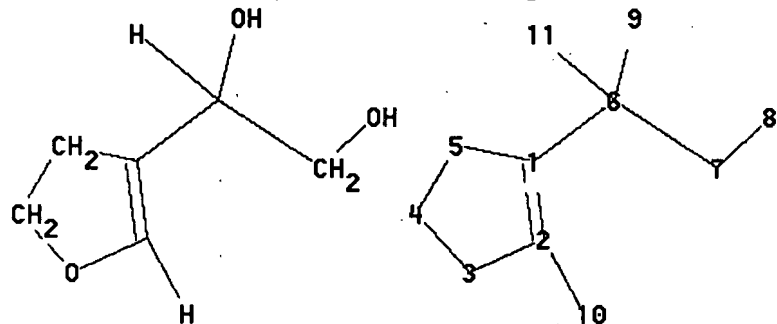


\*\*PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT\*\*

HOW MANY MORE ANSWERS DO YOU WISH TO SCAN? (1):end

=>

Uploading C:\Program Files\Stnexp\Queries\10517966B.str



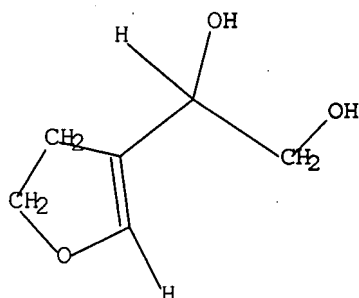
chain nodes :  
 6 7 8 9 10 11  
 ring nodes :

1 2 3 4 5  
 chain bonds :  
 1-6 2-10 6-7 6-9 6-11 7-8  
 ring bonds :  
 1-2 1-5 2-3 3-4 4-5  
 exact/norm bonds :  
 1-2 1-5 2-3 3-4 4-5 6-9  
 exact bonds :  
 1-6 2-10 6-7 6-11 7-8

Match level :  
 1:Atom 2:Atom 3:Atom 4:Atom 5:Atom 6:CLASS 7:CLASS 8:CLASS 9:CLASS  
 10:CLASS 11:CLASS

L4 STRUCTURE UPLOADED

=> d 14  
 L4 HAS NO ANSWERS  
 L4 STR



Structure attributes must be viewed using STN Express query preparation.

=> s sss 14 sam  
 SAMPLE SEARCH INITIATED 20:52:55 FILE 'REGISTRY'  
 SAMPLE SCREEN SEARCH COMPLETED - 863 TO ITERATE

100.0% PROCESSED 863 ITERATIONS 0 ANSWERS  
 SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS: ONLINE \*\*COMPLETE\*\*  
 BATCH \*\*COMPLETE\*\*  
 PROJECTED ITERATIONS: 15498 TO 19022  
 PROJECTED ANSWERS: 0 TO 0

L5 0 SEA SSS SAM L4

=> s sss 14 full  
 FULL SEARCH INITIATED 20:53:10 FILE 'REGISTRY'  
 FULL SCREEN SEARCH COMPLETED - 17644 TO ITERATE

100.0% PROCESSED 17644 ITERATIONS  
SEARCH TIME: 00.00.01

1 ANSWERS

L6 1 SEA SSS FUL L4

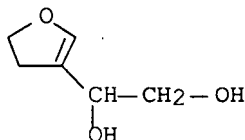
=> save temp doan10517966/a  
ENTER L#, L# RANGE, ALL, OR (END):end

=> save temp doan10517966/a l3  
ANSWER SET L3 HAS BEEN SAVED AS 'DOAN10517966/A'

=> save temp doal0517966/a l6  
ANSWER SET L6 HAS BEEN SAVED AS 'DOA10517966/A'

=> d scan l6

L6 1 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN  
IN 1,2-Ethanediol, 1-(4,5-dihydro-3-furanyl)- (9CI)  
MF C6 H10 O3



\*\*PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT\*\*

ALL ANSWERS HAVE BEEN SCANNED

=> l3 or l6  
L3 IS NOT A RECOGNIZED COMMAND  
The previous command name entered was not recognized by the system.  
For a list of commands available to you in the current file, enter  
"HELP COMMANDS" at an arrow prompt (=>).

=> s l3 or l6  
L7 29 L3 OR L6

=> fil caplus

COST IN U.S. DOLLARS	SINCE FILE ENTRY	TOTAL SESSION
FULL ESTIMATED COST	351.40	351.61

FILE 'CAPLUS' ENTERED AT 20:57:06 ON 17 AUG 2007  
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FILE COVERS 1907 - 17 Aug 2007 VOL 147 ISS 9  
FILE LAST UPDATED: 16 Aug 2007 (20070816/ED)

Effective October 17, 2005, revised CAS Information Use Policies apply. They are available for your review at:

<http://www.cas.org/infopolicy.html>

=> s 17

L8 67 L7

=> s 18 and (ay<2002 or py<2002 or pry<2002)

4176900 AY<2002

21892616 PY<2002

3653795 PRY<2002

L9 23 L8 AND (AY<2002 OR PY<2002 OR PRY<2002)

=> fil casreact

COST IN U.S. DOLLARS

SINCE FILE

TOTAL

ENTRY

SESSION

FULL ESTIMATED COST

8.38

359.99

FILE 'CASREACT' ENTERED AT 21:00:07 ON 17 AUG 2007  
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FILE CONTENT:1840 - 11 Aug 2007 VOL 147 ISS 8

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\*\*\*\*\*  
\* CASREACT now has more than 12 million reactions \*  
\* \*\*\*\*\*

Some CASREACT records are derived from the ZIC/VINITI database (1974-1999) provided by InfoChem, INPI data prior to 1986, and Biotransformations database compiled under the direction of Professor Dr. Klaus Kieslich.

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> s 17

L10 21 L7

=> s 123 or 110

L23 NOT FOUND

The L-number entered could not be found. To see the definition

of L-numbers, enter DISPLAY HISTORY at an arrow prompt (=>).

=> s 19 and 110

21 L7

73647 AY<2002

454899 PY<2002

26653 PRY<2002

L11 4 L9 AND L10

ENTER DISPLAY FORMAT (FCRDREF):all

L11 ANSWER 1 OF 4 CASREACT COPYRIGHT 2007 ACS on STN

AN 138:271663 CASREACT Full-text

TI Process for preparing intermediates for HIV aspartyl protease inhibitors, particularly (3 $\alpha$ ,3a $\beta$ ,6a $\beta$ )-hexahydrofuro[2,3-b]furan-3-ol and its (3R,3aS,6aR)-enantiomer

IN Doan, Brian Daniel; Davis, Roman D.; Lovelace, Thomas Claiborne

PA Smithkline Beecham Corporation, USA

SO PCT Int. Appl., 30 pp.

CODEN: PIXXD2

DT Patent

LA English

IC ICM C07D493-04

ICS C07D307-00; C07D309-00; C07D305-00

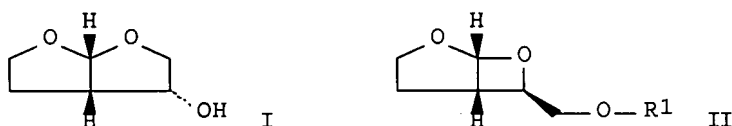
CC 28-2 (Heterocyclic Compounds (More Than One Hetero Atom))

Section cross-reference(s): 45

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 2003024974	A2	20030327	WO 2002-US29315	20020916
	WO 2003024974	A3	20040729		
	W:	AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW			
	RW:	GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG			
	AU 2002326925	A1	20030401	AU 2002-326925	20020916
	EP 1465897	A2	20041013	EP 2002-761678	20020916
	EP 1465897	B1	20060809		
	R:	AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, SK			
	JP 2005510467	T	20050421	JP 2003-528821	20020916
	AT 335745	T	20060915	AT 2002-761678	20020916
	ES 2265052	T3	20070201	ES 2002-2761678	20020916
	US 2004204595	A1	20041014	US 2004-490186	20040319
	US 7145024	B2	20061205		
PRAI	US 2001-323692P		20010920		
	WO 2002-US29315		20020916		
OS	MARPAT 138:271663				
GI					

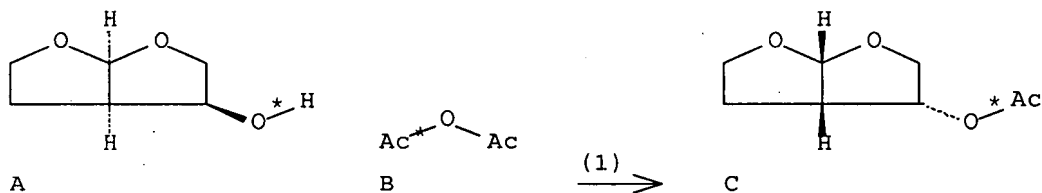




- AB The invention includes a method for preparing cyclic alcs. I (racemic or enantiomeric). The method involves a reduction, deprotection, and rearrangement, in non-aqueous telescoping conditions, of a bicyclic oxetane derivative II [R1 = C(R2)3, COR3, or Si(R3)3; R2 = (independently) H, alkyl, or aryl; R3 = (independently) alkyl or aryl]. The invention further provides a method of preparation of an intermediate useful in the synthesis of compds. that function as inhibitors of the aspartyl protease enzyme of human immunodeficiency virus (HIV). For instance, photochem. cycloaddn. of TBDMS-OCH2CHO with furan gave 98% yield of II [R1 = TBDMS, i.e., SiMe2Bu-tert]. The adduct underwent double-bond hydrogenation over water-wet 5% Pt/C in THF in the presence of K2CO3. This was followed (without isolation) by hydrolytic deprotection and rearrangement in THF solution in the presence of H2O and concentrated HCl, to give (+)-I in 82% yield (both steps). Racemic I was resolved by (1) O-acetylation with Ac2O, Na2CO3, and DMAP; (2) selective hydrolysis of the undesired enantiomer of the acetate using the lipase PS-800 in phosphate buffer at pH 6.8-7.2, giving the (3R,3aS,6aR)-acetate in >98% ee; and (3) hydrolysis using K2CO3 in MeOH at room temperature, giving (3R,3aS,6aR)-I. Other protecting groups for use in R1, namely PhCM2, tert-Bu, and PhCH2, are exemplified.
- ST hexahydrofurofuranol prepn intermediate HIV aspartyl protease inhibitor; dioxabicycloheptenemethanol prepn hydrogenation rearrangement hydrolysis; photochem cycloaddn furan protected hydroxyacetaldehyde
- IT Cycloaddition reaction  
 ([2+2], photochem., of furan with protected hydroxyacetaldehydes; preparation of hexahydrofurofuranol racemate and enantiomer as intermediates  
 for HIV aspartyl protease inhibitors)
- IT Protective groups  
 (deprotection of protected dioxabicycloheptanemethanol derivs.; preparation of hexahydrofurofuranol racemate and enantiomer as intermediates for HIV aspartyl protease inhibitors)
- IT Rearrangement  
 (of dioxabicycloheptanemethanol derivs.; preparation of hexahydrofurofuranol  
 racemate and enantiomer as intermediates for HIV aspartyl protease inhibitors)
- IT Hydrogenation  
 (of dioxabicycloheptenemethanol derivs.; preparation of hexahydrofurofuranol  
 racemate and enantiomer as intermediates for HIV aspartyl protease inhibitors)
- IT Anti-AIDS agents  
 (preparation of hexahydrofurofuranol racemate and enantiomer as intermediates for HIV aspartyl protease inhibitors)
- IT 162020-29-3P, (3S,3aR,6aS)-Hexahydrofuro[2,3-b]furan-3-yl acetate  
 RL: BSU (Biological study, unclassified); BYP (Byproduct); RCT (Reactant); BIOL (Biological study); PREP (Preparation); RACT (Reactant or reagent)  
 (byproduct enantiomer; preparation of hexahydrofurofuranol racemate and enantiomer as intermediates for HIV aspartyl protease inhibitors)
- IT 144114-21-6, HIV aspartyl protease  
 RL: MSC (Miscellaneous)  
 (preparation of hexahydrofurofuranol racemate and enantiomer as

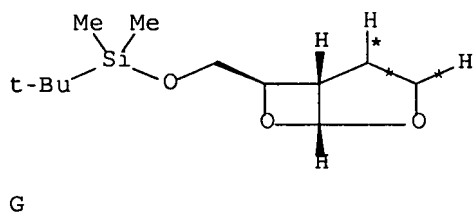
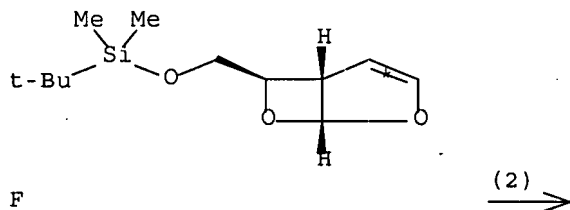
- intermediates for HIV aspartyl protease inhibitors)
- IT 162119-35-9P, (3R,3aS,6aR)-Hexahydrofuro[2,3-b]furan-3-yl acetate  
 RL: BMF (Bioindustrial manufacture); BPN (Biosynthetic preparation); IMF (Industrial manufacture); PUR (Purification or recovery); RCT (Reactant); SPN (Synthetic preparation); BIOL (Biological study); PREP (Preparation); RACT (Reactant or reagent)  
 (process intermediate; preparation of hexahydrofurofuranol racemate and enantiomer as intermediates for HIV aspartyl protease inhibitors)
- IT 186488-43-7P, (3 $\alpha$ ,3a $\beta$ ,6a $\beta$ )-Hexahydrofuro[2,3-b]furan-3-yl acetate  
 RL: BSU (Biological study, unclassified); IMF (Industrial manufacture); RCT (Reactant); SPN (Synthetic preparation); BIOL (Biological study); PREP (Preparation); RACT (Reactant or reagent)  
 (process intermediate; preparation of hexahydrofurofuranol racemate and enantiomer as intermediates for HIV aspartyl protease inhibitors)
- IT 130827-15-5P, (3 $\beta$ ,3a $\beta$ ,6a $\beta$ )-[(2,7-Dioxabicyclo[3.2.0]hept-3-en-6-yl)methoxy]-tert-butyldimethylsilane 503189-87-5P,  
 (3 $\beta$ ,3a $\beta$ ,6a $\beta$ )-[(2,7-Dioxabicyclo[3.2.0]hept-6-yl)methoxy]-tert-butyldimethylsilane 503189-88-6P, (3 $\beta$ ,3a $\beta$ ,6a $\beta$ )-6-[(1-Methyl-1-phenylethoxy)methyl]-2,7-dioxabicyclo[3.2.0]hept-3-ene 503189-89-7P, (3 $\beta$ ,3a $\beta$ ,6a $\beta$ )-6-[(1-Methyl-1-phenylethoxy)methyl]-2,7-dioxabicyclo[3.2.0]heptane 503189-90-0P, (3 $\beta$ ,3a $\beta$ ,6a $\beta$ )-6-(tert-Butoxymethyl)-2,7-dioxabicyclo[3.2.0]hept-3-ene 503189-91-1P, (3 $\beta$ ,3a $\beta$ ,6a $\beta$ )-6-(tert-Butoxymethyl)-2,7-dioxabicyclo[3.2.0]heptane 503189-92-2P, (3 $\beta$ ,3a $\beta$ ,6a $\beta$ )-6-[(Benzyloxy)methyl]-2,7-dioxabicyclo[3.2.0]hept-3-ene 503189-93-3P, (1-Methyl-1-phenylethoxy)acetaldehyde 503189-94-4P  
 RL: IMF (Industrial manufacture); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
 (process intermediate; preparation of hexahydrofurofuranol racemate and enantiomer as intermediates for HIV aspartyl protease inhibitors)
- IT 100-58-3, Phenylmagnesium bromide 100-79-8, (+)-2,2-Dimethyl-1,3-dioxolane-4-methanol 110-00-9, Furan 28047-97-4, tert-Butoxyacetaldehyde 60656-87-3, (Benzyloxy)acetaldehyde 102191-92-4, [(tert-Butyldimethylsilyl)oxy]acetaldehyde  
 RL: RCT (Reactant); RACT (Reactant or reagent)  
 (starting material; preparation of hexahydrofurofuranol racemate and enantiomer as intermediates for HIV aspartyl protease inhibitors)
- IT 109789-19-7P, Hexahydrofuro[2,3-b]furan-3-ol  
 RL: BMF (Bioindustrial manufacture); BPN (Biosynthetic preparation); IMF (Industrial manufacture); PUR (Purification or recovery); RCT (Reactant); SPN (Synthetic preparation); BIOL (Biological study); PREP (Preparation); RACT (Reactant or reagent)  
 (target intermediate; preparation of hexahydrofurofuranol racemate and enantiomer as intermediates for HIV aspartyl protease inhibitors)
- IT 156928-09-5P, (3R,3aS,6aR)-Hexahydrofuro[2,3-b]furan-3-ol  
 RL: BMF (Bioindustrial manufacture); BPN (Biosynthetic preparation); IMF (Industrial manufacture); PUR (Purification or recovery); SPN (Synthetic preparation); BIOL (Biological study); PREP (Preparation)  
 (target intermediate; preparation of hexahydrofurofuranol racemate and enantiomer as intermediates for HIV aspartyl protease inhibitors)
- IT 162119-33-7P, (3 $\alpha$ ,3a $\beta$ ,6a $\beta$ )-Hexahydrofuro[2,3-b]furan-3-ol  
 RL: IMF (Industrial manufacture); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
 (target intermediate; preparation of hexahydrofurofuranol racemate and enantiomer as intermediates for HIV aspartyl protease inhibitors)

RX(1) OF 77 ...A + B ==> C...



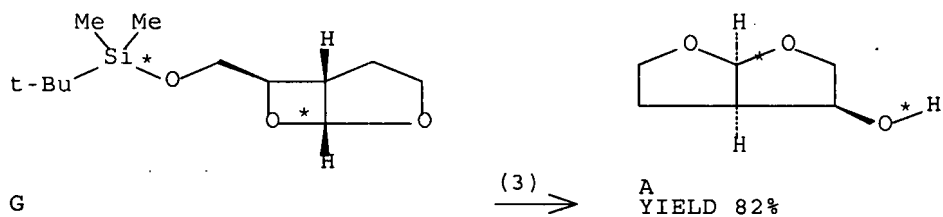
RX(1)  
 RCT A 162119-33-7, B 108-24-7  
 RGT D 497-19-8 Na<sub>2</sub>CO<sub>3</sub>  
 PRO C 186488-43-7  
 CAT 1122-58-3 4-DMAP  
 CON SUBSTAGE(1) <10 deg C  
 SUBSTAGE(2) 10 deg C -> room temperature  
 SUBSTAGE(3) overnight, room temperature  
 NTE scalable

RX(2) OF 77 ...F ==> G...



RX(2)  
 RCT F 130827-15-5  
 RGT H 584-08-7 K<sub>2</sub>CO<sub>3</sub>, I 1333-74-0 H<sub>2</sub>  
 PRO G 503189-87-5  
 CAT 7440-06-4 Pt  
 SOL 109-99-9 THF, 7732-18-5 Water  
 CON overnight, room temperature, 0.26 bar  
 NTE scalable

RX(3) OF 77 ...G ==> A...



RX(3) RCT G 503189-87-5

STAGE(1)

RGT M 7647-01-0 HCl  
 SOL 109-99-9 THF, 7732-18-5 Water  
 CON 1 hour, room temperature

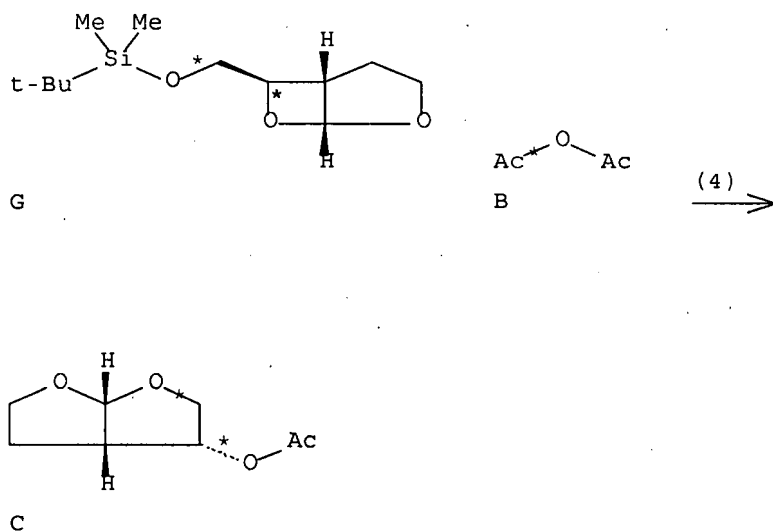
STAGE(2)

RGT H 584-08-7 K<sub>2</sub>CO<sub>3</sub>  
 CON pH 7

PRO A 162119-33-7

NTE scalable

RX(4) OF 77 ...G + B ==> C...



RX(4) RCT G 503189-87-5

STAGE(1)

RGT M 7647-01-0 HCl  
 SOL 109-99-9 THF, 7732-18-5 Water  
 CON 1 hour, room temperature

STAGE(2)

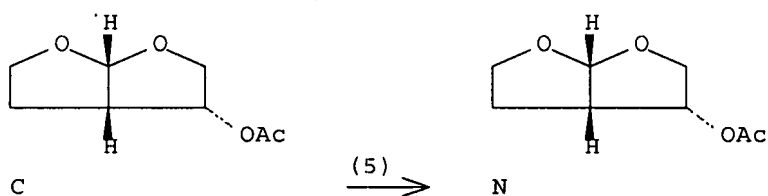
RGT H 584-08-7 K<sub>2</sub>CO<sub>3</sub>  
 CON pH 7

STAGE(3)

RCT B 108-24-7  
 RGT D 497-19-8 Na<sub>2</sub>CO<sub>3</sub>  
 CAT 1122-58-3 4-DMAP  
 CON SUBSTAGE(1) overnight, room temperature  
 SUBSTAGE(2) 3 hours, room temperature

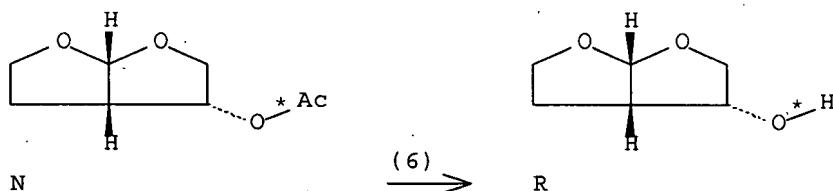
PRO C 186488-43-7  
 NTE scalable

RX(5) OF 77 ...C ==> N...



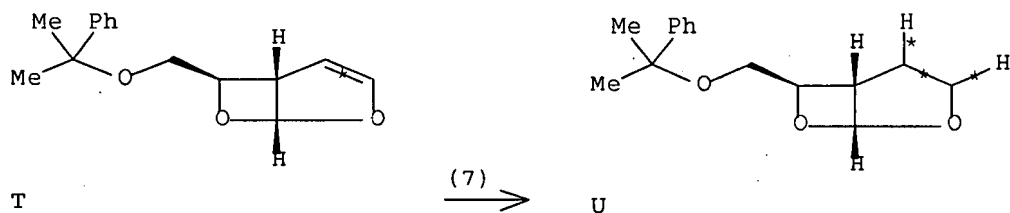
RX(5) RCT C 186488-43-7  
 RGT O 7558-80-7 NaH<sub>2</sub>PO<sub>4</sub>, P 1310-73-2 NaOH  
 PRO N 162119-35-9  
 CAT 9001-62-1 Lipase  
 CON SUBSTAGE(1) room temperature -> 35 deg C  
 SUBSTAGE(2) pH 7.0  
 SUBSTAGE(3) pH 6.8 - 7.2  
 NTE scalable, PS-800 lipase, >98% ee product

RX(6) OF 77 ...N ==> R



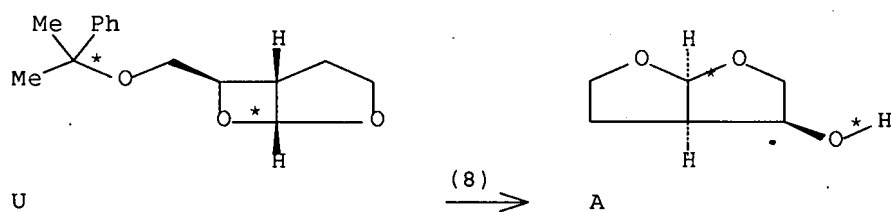
RX(6) RCT N 162119-35-9  
 RGT H 584-08-7 K<sub>2</sub>CO<sub>3</sub>  
 PRO R 156928-09-5  
 SOL 67-56-1 MeOH  
 CON 18 - 20 hours, room temperature  
 NTE scalable

RX(7) OF 77 ...T ==> U...



RX(7)    RCT   T 503189-88-6  
          RGT   H 584-08-7 K<sub>2</sub>CO<sub>3</sub>, I 1333-74-0 H<sub>2</sub>  
          PRO   U 503189-89-7  
          CAT   7440-06-4 Pt  
          SOL   109-99-9 THF, 7732-18-5 Water  
          CON   overnight, room temperature, 0.26 bar  
          NTE   scalable

RX(8) OF 77    ...U ==> A...



RX(8)    RCT   U 503189-89-7

STAGE(1)

RGT   M 7647-01-0 HCl  
 SOL   109-99-9 THF, 7732-18-5 Water  
 CON   1 hour, room temperature

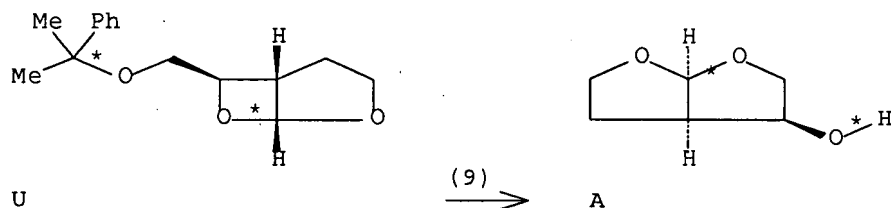
STAGE(2)

RGT   H 584-08-7 K<sub>2</sub>CO<sub>3</sub>  
 CON   pH 7

PRO   A 162119-33-7

NTE   scalable

RX(9) OF 77    U ==> A



RX(9) RCT U 503189-89-7

STAGE(1)

CAT 53664-47-4 Amberlyst  
SOL 109-99-9 THF, 7732-18-5 Water  
CON 1 hour, room temperature

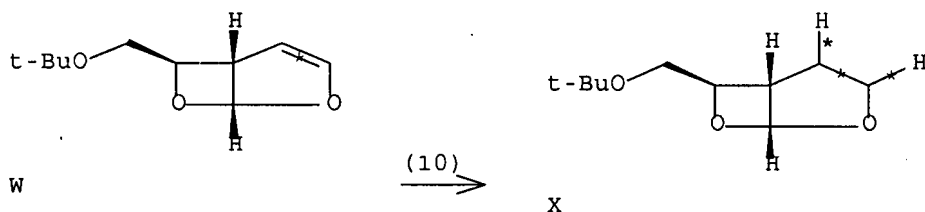
STAGE(2)

RGT H 584-08-7 K<sub>2</sub>CO<sub>3</sub>  
CON pH 7

PRO A 162119-33-7

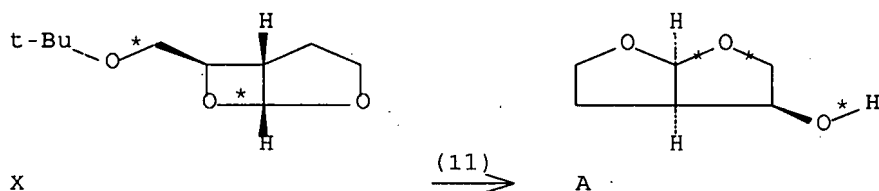
NTE scalable

RX(10) OF 77 ...W ==> X...



RX(10) RCT W 503189-90-0  
RGT H 584-08-7 K<sub>2</sub>CO<sub>3</sub>, I 1333-74-0 H<sub>2</sub>  
PRO X 503189-91-1  
CAT 7440-06-4 Pt  
SOL 109-99-9 THF, 7732-18-5 Water  
CON overnight, room temperature, 0.26 bar  
NTE scalable

RX(11) OF 77 ...X ==> A...



RX(11) RCT X 503189-91-1

STAGE(1)

RGT Y 76-05-1 F<sub>3</sub>CCO<sub>2</sub>H  
SOL 75-89-8 F<sub>3</sub>CCH<sub>2</sub>OH, 7732-18-5 Water  
CON 1 hour, room temperature

STAGE(2)

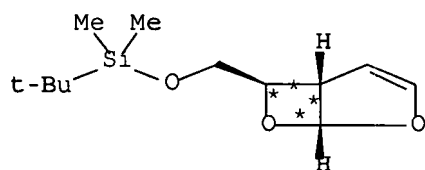
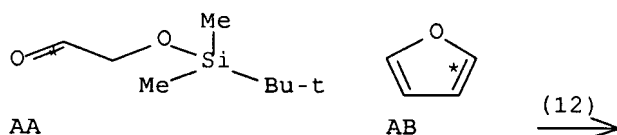
RGT H 584-08-7 K<sub>2</sub>CO<sub>3</sub>

CON pH 7

PRO A 162119-33-7

NTE scalable

RX(12) OF 77 AA + AB ==> F...



F  
YIELD 98%

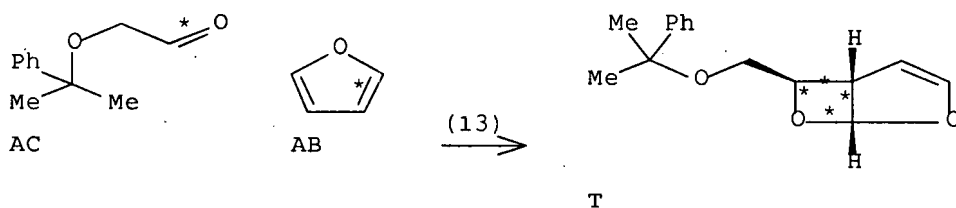
RX(12) RCT AA 102191-92-4, AB 110-00-9

PRO F 130827-15-5

CON 48 hours

NTE photochem., cooling, flow system, scalable

RX(13) OF 77 ...AC + AB ==> T...



RX(13) RCT AC 503189-93-3, AB 110-00-9

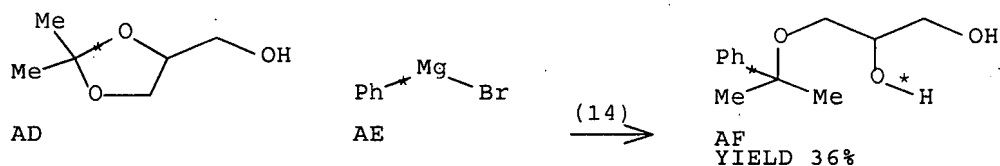
PRO T 503189-88-6

CON 48 hours

NTE photochem., cooling, flow system, scalable

RX(14) OF 77 AD + AE ==> AF...





RX(14) RCT AD 100-79-8, AE 100-58-3

STAGE(1)

SOL 1330-20-7 Xylene, 109-99-9 THF

CON SUBSTAGE(1) room temperature -> 100 deg C

SUBSTAGE(2) 42 hours, 100 deg C

SUBSTAGE(3) 100 deg C -> 30 deg C

STAGE(2)

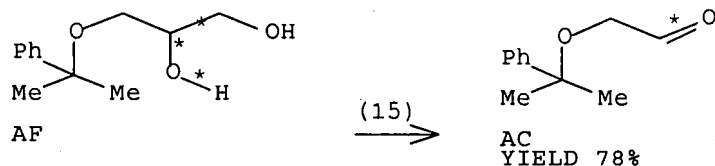
RGT AG 7778-77-0 KH<sub>2</sub>PO<sub>4</sub>

SOL 7732-18-5 Water

PRO AF 503189-94-4

NTE scalable

RX(15) OF 77 ...AF ==> AC...



RX(15) RCT AF 503189-94-4

PRO AC 503189-93-3

SOL 75-09-2 CH<sub>2</sub>Cl<sub>2</sub>, 7732-18-5 Water, 7790-28-5 NaIO<sub>4</sub>

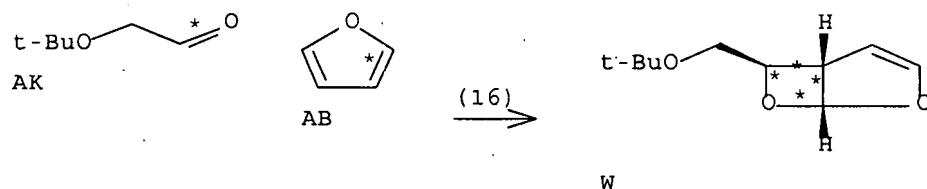
CON SUBSTAGE(1) 0 deg C

SUBSTAGE(2) 0 deg C -> 40 deg C

SUBSTAGE(3) 90 minutes

NTE presence of silica gel, scalable

RX(16) OF 77 AK + AB ==> W...



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=> s 18

L9 1 L8

=> s 18 or 19

1 L8

L10 1 L8 OR L9

=> s 18 and 19

1 L8

L11 1 L8 AND L9

=> s 16 and 19

L12 1 L6 AND L9

=> s 16 or 19

L13 2 L6 OR L9

=> d ibib abs hitstr 1-2

L13 ANSWER 1 OF 2 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 2005:14172 CAPLUS Full-text

DOCUMENT NUMBER: 142:114047

TITLE: A preparation of furofuranyl derivative, useful as inhibitor of HIV aspartyl protease

INVENTOR(S): Roberts, John Charles; Toczko, Jennifer Fell

PATENT ASSIGNEE(S): SmithKline Beecham Corporation, USA; Martin, Michael Tolar

SOURCE: PCT Int. Appl., 36 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
------------	------	------	-----------------	------

WO 2005000249	A2	20050106	WO 2004-US20353	20040625
WO 2005000249	A3	20050407		
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW				
RW: BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
EP 1638960	A2	20060329	EP 2004-777060	20040625
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, CY, TR, BG, CZ, EE, HU, PL, SK, HR				
JP 2007521277	T	20070802	JP 2006-517643	20040625
US 2006148865	A1	20060706	US 2005-560500	20051212
PRIORITY APPLN. INFO.:			US 2003-483002P	P 20030627
			WO 2004-US20353	W 20040625
OTHER SOURCE(S):		CASREACT 142:114047		
GI				

\* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT \*

AB The invention relates to a preparation of furofuranyl derivative I, useful as inhibitor of HIV aspartyl protease (no biol. data). For instance, I was prepared via deprotection of II and coupling with III with a yield of 90% (example 2).

IT 640289-31-2P

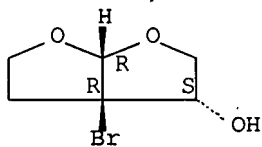
RL: IMF (Industrial manufacture); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(preparation of furofuranyl derivative useful as inhibitor of HIV aspartyl protease)

RN 640289-31-2 CAPLUS

CN Furo[2,3-b]furan-3-ol, 3a-bromohexahydro-, (3R,3aS,6aS)-rel- (9CI) (CA INDEX NAME)

Relative stereochemistry.



L13 ANSWER 2 OF 2 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 2004:20676 CAPLUS Full-text

DOCUMENT NUMBER: 140:77015

TITLE: Preparation of stereoisomers of  
3 $\alpha$ ,3 $\alpha\beta$ ,6 $\alpha\beta$ -hexahydrofuro[2,3-b]furan-3-ol

INVENTOR(S): Doan, Brian Daniel; Patterson, Daniel Edward; Roberts, John C.  
 PATENT ASSIGNEE(S): Smithkline Beecham Corporation, USA  
 SOURCE: PCT Int. Appl., 53 pp.  
 CODEN: PIXXD2  
 DOCUMENT TYPE: Patent  
 LANGUAGE: English  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

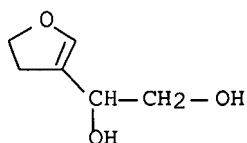
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2004002975	A1	20040108	WO 2003-US20094	20030625
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
AU 2003247651	A1	20040119	AU 2003-247651	20030625
EP 1532127	A1	20050525	EP 2003-762054	20030625
EP 1532127	B1	20060927		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, SK				
JP 2005533821	T	20051110	JP 2004-517842	20030625
AT 340788	T	20061015	AT 2003-762054	20030625
ES 2268427	T3	20070316	ES 2003-3762054	20030625
US 2005261507	A1	20051124	US 2004-517966	20041214
PRIORITY APPLN. INFO.:			US 2002-392677P	P 20020627
			WO 2003-US20094	W 20030625

AB A process for the preparation of stereoisomers of 3 $\alpha$ ,3 $\alpha\beta$ ,6 $\alpha\beta$ -hexahydrofuro[2,3-b]furan-3-ol is disclosed. For instance, treatment of 2,3-dihydrofuran with Et chlorooxoacetate (MTBE, Et3N) provides Et  $\alpha$ -oxo-4,5-dihydrofuran-3-ylacetate as an oil which is reduced to the diol (THF, LAH) and cyclized (THF/H2O, NBS) to give 3 $\alpha$ -bromohexahydrofuro[2,3-b]furan-3-ol as a mixture of 2 diastereomers (3:1). This is reduced (THF, Et3N, H2-Pd/C) and acetylated to give acetic acid hexahydrofuro[2,3-b]furan-3-yl ester. Minor isomer acetates are reacted with a lipase (0.1N Na2HPO4, pH 7.0, 35°, PS-800) and the unreacted acetate starting material (organic extract) is deacylated (MeOH, K2CO3) to give 3R,3aS,6aR-hexahydrofuro[2,3-b]furan-3-ol. Preparation of 3 $\alpha$ -bromo analogs are also described. Compds. disclosed herein are useful in the preparation of compds. that may be inhibitors of HIV aspartyl protease. The current process uses inexpensive, nonchiral starting materials and does not rely on heavy metals or photochem. compared to prior art methods.

IT 640289-32-3P, 1-(4,5-Dihydrofuran-3-yl)ethane-1,2-diol  
 640289-33-4P, 3 $\alpha$ -Bromohexahydrofuro[2,3-b]furan-3-ol  
 RL: IMF (Industrial manufacture); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
 (preparation of stereoisomers of 3 $\alpha$ ,3 $\alpha\beta$ ,6 $\alpha\beta$ -hexahydrofuro[2,3-b]furan-3-ol via 2,3-dihydrofuran annulation and enzymic resolution)

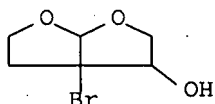
RN 640289-32-3 CAPLUS

CN 1,2-Ethanediol, 1-(4,5-dihydro-3-furanyl)- (9CI) (CA INDEX NAME)



RN 640289-33-4 CAPLUS

CN Furo[2,3-b]furan-3-ol, 3a-bromohexahydro- (9CI) (CA INDEX NAME)



IT 640289-30-1P, (3S,3AR,6aR)-3a-bromohexahydrofuro[2,3-b]furan-3-ol  
640289-31-2P, rel-(3S,3AR,6aR)-3a-bromohexahydrofuro[2,3-b]furan-3-ol

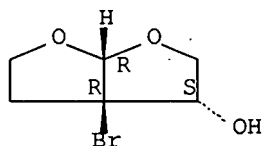
RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)

(preparation of stereoisomers of 3 $\alpha$ ,3a $\beta$ ,6a $\beta$ -hexahydrofuro[2,3-b]furan-3-ol via 2,3-dihydrofuran annulation and enzymic resolution)

RN 640289-30-1 CAPLUS

CN Furo[2,3-b]furan-3-ol, 3a-bromohexahydro-, (3S,3aR,6aR)- (9CI) (CA INDEX NAME)

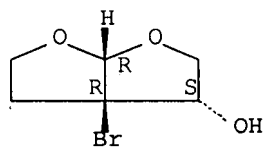
Absolute stereochemistry.



RN 640289-31-2 CAPLUS

CN Furo[2,3-b]furan-3-ol, 3a-bromohexahydro-, (3R,3aS,6aS)-rel- (9CI) (CA INDEX NAME)

Relative stereochemistry.



REFERENCE COUNT: 7 THERE ARE 7 CITED REFERENCES AVAILABLE FOR THIS  
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	ENTRY	SESSION
CA SUBSCRIBER PRICE	-1.56	-1.56

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FILE CONTENT:1840 - 11 Aug 2007 VOL 147 ISS 8

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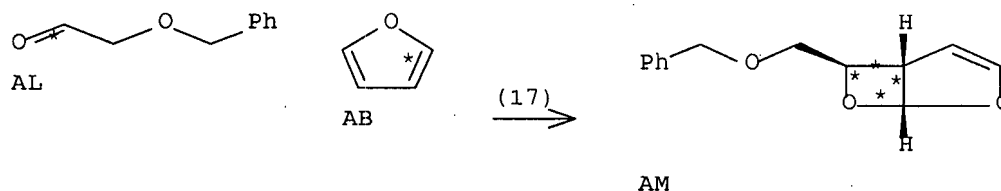
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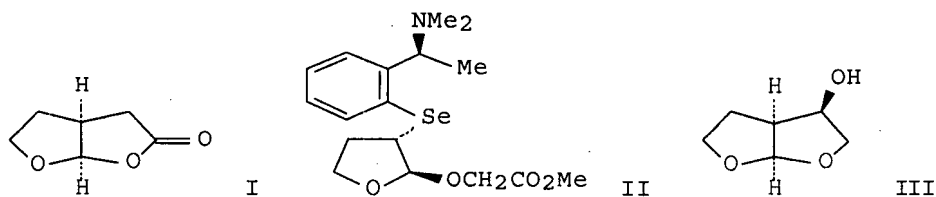
RX(16) RCT AK 28047-97-4, AB 110-00-9  
 PRO W 503189-90-0  
 CON 48 hours  
 NTE photochem., cooling, flow system, scalable

RX(17) OF 77 AL + AB ==> AM



RX(17) RCT AL 60656-87-3, AB 110-00-9  
 PRO AM 503189-92-2  
 CON 48 hours  
 NTE photochem., cooling, flow system, scalable

L11 ANSWER 2 OF 4 CASREACT COPYRIGHT 2007 ACS on STN  
 AN 135:273092 CASREACT [Full-text](#)  
 TI Stereoselective synthesis of optically active perhydrofuro[2,3-b]furan derivatives  
 AU Uchiyama, M.; Hirai, M.; Nagata, M.; Katoh, R.; Ogawa, R.; Ohta, A.  
 CS School of Pharmacy, Tokyo University of Pharmacy and Life Science, Hachioji, Tokyo, 192-0392, Japan  
 SO Tetrahedron Letters (2001), 42(28), 4653-4656  
 CODEN: TELEAY; ISSN: 0040-4039  
 PB Elsevier Science Ltd.  
 DT Journal  
 LA English  
 CC 30-20 (Terpenes and Terpenoids)  
 GI



AB (1R,5S)-2,8-Dioxabicyclo[3.3.0]octan-3-one (I) and its derivs., important subunits in various biol. active natural products, were synthesized based on a new approach using the asym. oxyselenenylation of 2,3-dihydrofuran as the key step yielding II which was cyclized and resolved providing the major isomer III.  
 ST dioxabicyclooctanone prepn abs configuration; cyclization radical selenenylation oxy furan deriv; furofuran deriv prepn abs configuration;



oxyselenenylation asym dihydrofuran

IT Heterocyclic compounds  
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
 (bicyclic, oxa-; preparation of optically active perhydrofuro[2,3-b]furan derivs.)

IT Bicyclic compounds  
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
 (heterocyclic, oxa-; preparation of optically active perhydrofuro[2,3-b]furan derivs.)

IT Absolute configuration  
 (of perhydrofuro[2,3-b]furan derivs.)

IT Cyclization  
 (radical; of oxyselenenylated furan derivative)

IT Substitution reaction  
 (selenenylation, asym. oxy-; of 2,3-dihydrofuran)

IT 174590-88-6P  
 RL: PNU (Preparation, unclassified); PREP (Preparation)  
 (stereoselective preparation of optically active perhydrofuro[2,3-b]furan derivs.)

IT 96-35-5, Methyl glycolate 1191-99-7, 2,3-Dihydrofuran 42340-98-7,  
 (R)-1-(1-Naphthyl)ethyl isocyanate 172842-01-2  
 RL: RCT (Reactant); RACT (Reactant or reagent)  
 (stereoselective preparation of optically active perhydrofuro[2,3-b]furan derivs.)

IT 156928-09-5P 252873-50-0P 362634-52-4P 362634-60-4P 362634-62-6P  
 362634-64-8P 362634-66-0P  
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
 (stereoselective preparation of optically active perhydrofuro[2,3-b]furan derivs.)

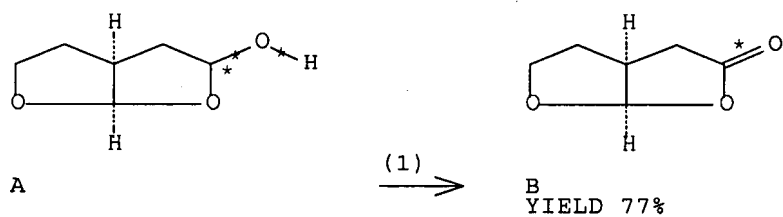
IT 152185-61-0P 156928-10-8P 362634-54-6P 362634-56-8P  
 RL: SPN (Synthetic preparation); PREP (Preparation)  
 (stereoselective preparation of optically active perhydrofuro[2,3-b]furan derivs.)

RE.CNT 20 THERE ARE 20 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

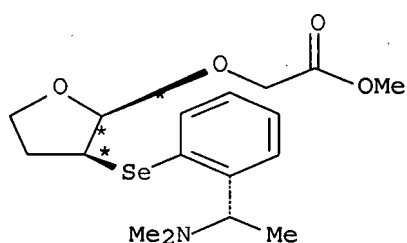
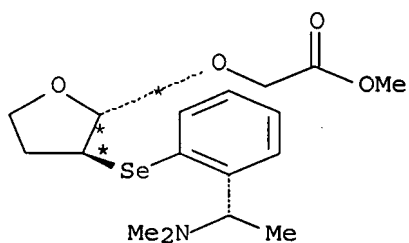
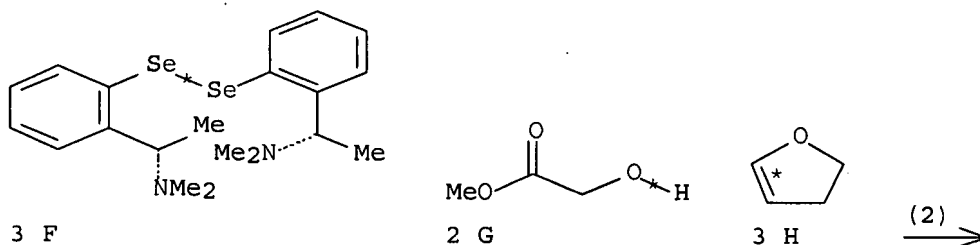
- (1) Barton, D; J Chem Soc 1961, P5061 CAPLUS
- (2) Chen, X; Bioorg Med Chem Lett 1996, V6, P2847 CAPLUS
- (3) Enders, D; J Chem Soc, Chem Commun 1999, P701 CAPLUS
- (4) Ghosh, A; J Med Chem 1994, V37, P2506 CAPLUS
- (5) Ghosh, A; Tetrahedron Lett 1995, V36, P505 CAPLUS
- (6) Hochlowski, J; J Org Chem 1983, V48, P1141 CAPLUS
- (7) Hosozawa, S; Phytochemistry 1973, V12, P1833 CAPLUS
- (8) Jalali, M; Tetrahedron Lett 1983, V24, P4307 CAPLUS
- (9) Merritt, A; Nat Prod Rep 1992, P243 CAPLUS
- (10) Petit, F; Synthesis 1995, P1517 CAPLUS
- (11) Petit, F; Tetrahedron: Asymmetry 1993, V4, P1341 CAPLUS
- (12) Pirkle, W; J Org Chem 1974, V39, P3904 CAPLUS
- (13) Pirkle, W; J Org Chem 1977, V42, P1370 CAPLUS
- (14) Pirkle, W; J Org Chem 1977, V42, P384 CAPLUS
- (15) Schuda, P; Top Curr Chem 1980, V91, P75 CAPLUS
- (16) Takizawa, P; Cell 1993, V73, P1079 CAPLUS
- (17) Uchiyama, M; Tetrahedron Lett 2001, V42, P1559 CAPLUS
- (18) Uchiyama, M; Tetrahedron Lett 2001, V42, P1931 CAPLUS
- (19) Vader, J; Tetrahedron 1989, V45, P2131 CAPLUS
- (20) van Beeck, T; Recl Trav Chim Pays-Bas 1986, V105, P513

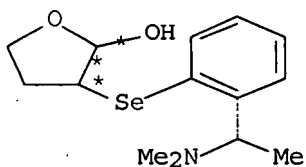
RX(1) OF 44 ...A ==> B



RX(1) RCT A 362634-66-0  
RGT C 26299-14-9 PCC, D 127-09-3 AcONa  
PRO B 152185-61-0  
SOL 75-09-2 CH<sub>2</sub>Cl<sub>2</sub>  
NTE mol. sieves used

RX(2) OF 44 3 F + 2 G + 3 H ==> I + J + K...





K  
YIELD 15%

RX(2) RCT F 172842-01-2

STAGE(1)

RGT L 7791-25-5 SO2Cl2

SOL 75-09-2 CH2Cl2

STAGE(2)

RGT M 14104-20-2 AgBF4

SOL 75-09-2 CH2Cl2

STAGE(3)

RCT G 96-35-5

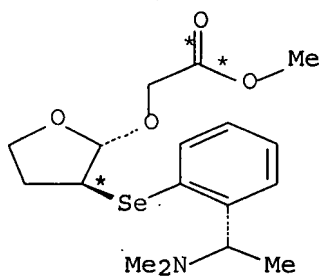
STAGE(4)

RCT H 1191-99-7

PRO I 362634-52-4, J 362634-54-6, K 362634-56-8

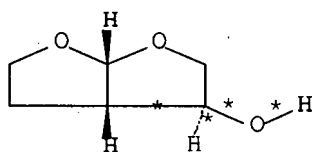
NTE stereoselective, optimized on temp., key step

RX(3) OF 44 ...2 I ==> N + O...

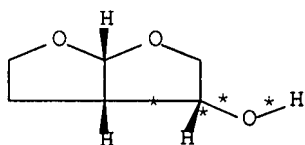


2 I

(3) →



N  
YIELD 13%



O  
YIELD 44%

RX(3) RCT I 362634-52-4

STAGE(1)

RGT P 1191-15-7  $\text{AlH}(\text{Bu-i})_2$

SOL 108-88-3 PhMe

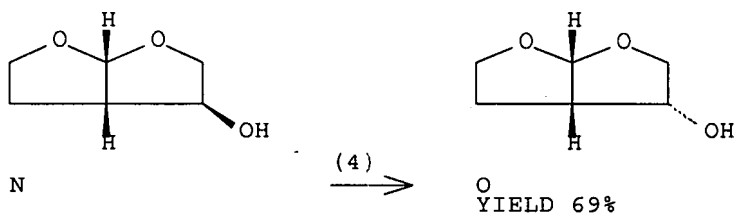
STAGE(2)

RGT Q 688-73-3  $\text{Bu}_3\text{SnH}$ , R 78-67-1 AIBN

SOL 71-43-2 Benzene

PRO N 252873-50-0, O 156928-09-5

RX(4) OF 44 ...N ==> O...



RX(4) RCT N 252873-50-0

STAGE(1)

RGT C 26299-14-9 PCC

SOL 75-09-2  $\text{CH}_2\text{Cl}_2$

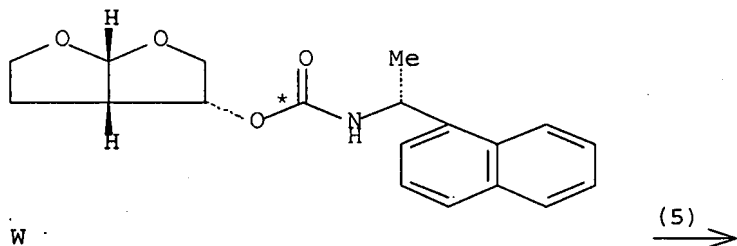
STAGE(2)

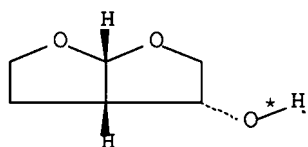
RGT U 16940-66-2  $\text{NaBH}_4$

SOL 64-17-5 EtOH

PRO O 156928-09-5

RX(5) OF 44 W ==> O...

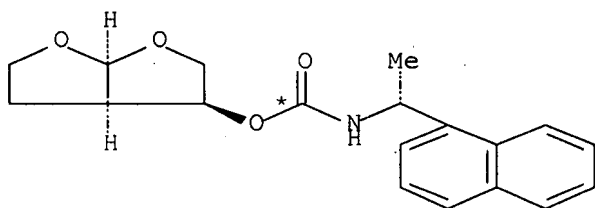




O  
YIELD 86%

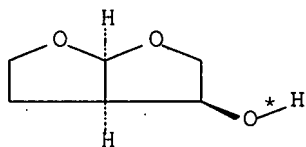
RX(5)      RCT   W 362634-60-4  
              RGT   X 16853-85-3 LiAlH<sub>4</sub>  
              PRO   O 156928-09-5  
              SOL   109-99-9 THF

RX(6) OF 44      ...Z ==> AA



Z

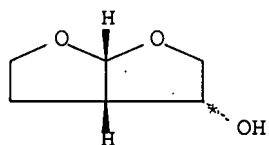
(6) →



AA  
YIELD 78%

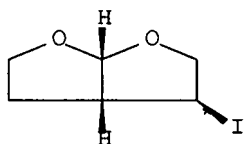
RX(6)      RCT   Z 362634-62-6  
              RGT   X 16853-85-3 LiAlH<sub>4</sub>  
              PRO   AA 156928-10-8  
              SOL   109-99-9 THF

RX(7) OF 44      ...O ==> AB...



O

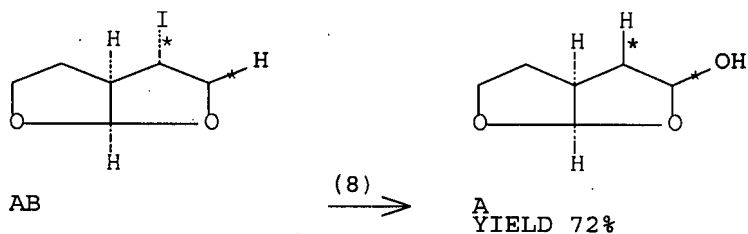
(7) →



AB  
YIELD 87%

RX(7) RCT O 156928-09-5  
RGT AC 603-35-0 PPh<sub>3</sub>, AD 288-32-4 1H-Imidazole, AE 7553-56-2 I<sub>2</sub>  
PRO AB 362634-64-8  
SOL 71-43-2 Benzene

RX(8) OF 44 ...AB ==> A...



RX(8) RCT AB 362634-64-8

STAGE(1)

RGT AF 6674-22-2 DBU

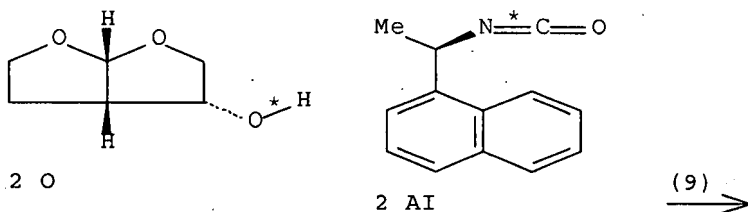
STAGE(2)

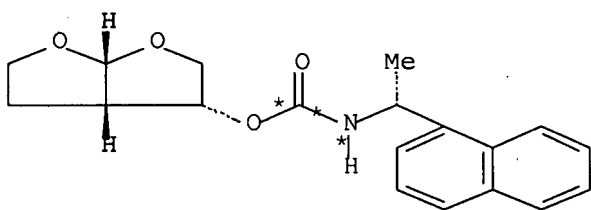
RGT AG 7664-93-9 H<sub>2</sub>SO<sub>4</sub>

SOL 109-99-9 THF, 7732-18-5 Water

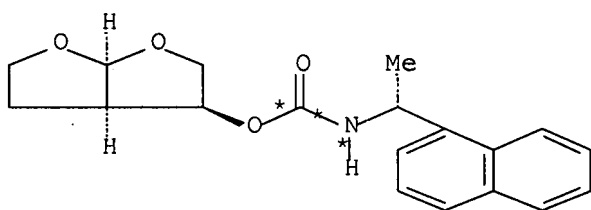
PRO A 362634-66-0

RX(9) OF 44 ...2 O + 2 AI ==> W + Z...





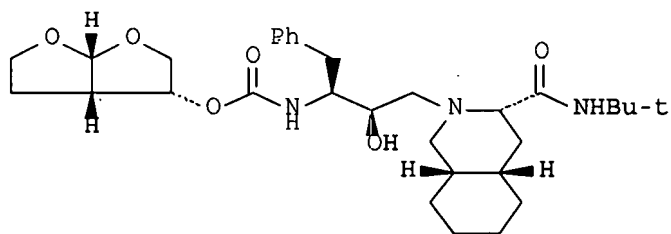
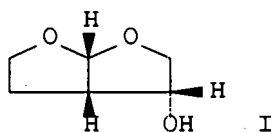
W  
YIELD 73%



Z  
YIELD 22%

RX(9) RCT O 156928-09-5, AI 42340-98-7  
RGT AJ 108-01-0 Me2NCH2CH2OH  
PRO W 362634-60-4, Z 362634-62-6  
SOL 71-43-2 Benzene  
NTE stereoselective

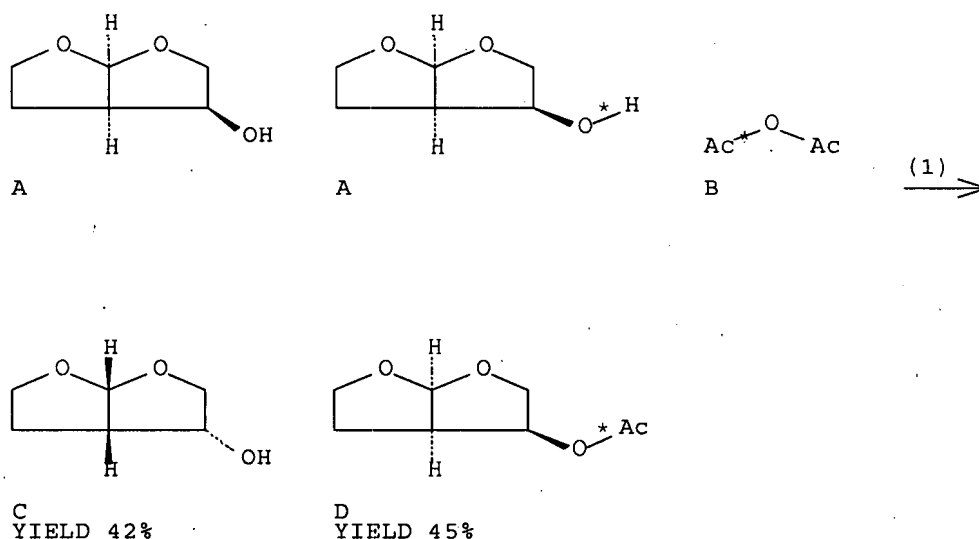
L11 ANSWER 3 OF 4 CASREACT COPYRIGHT 2007 ACS on STN  
AN 122:239645 CASREACT Full-text  
TI Synthesis and optical resolution of high affinity P2-ligands for HIV-1  
protease inhibitors  
AU Ghosh, Arun K.; Chen, Yan  
CS Dept. Chem., Univ. Illinois at Chicago, Chicago, IL, 60607, USA  
SO Tetrahedron Letters (1995), 36(4), 505-8  
CODEN: TELEAY; ISSN: 0040-4039  
PB Elsevier  
DT Journal  
LA English  
CC 28-18 (Heterocyclic Compounds (More Than One Hetero Atom))  
Section cross-reference(s): 27, 29  
GI



II

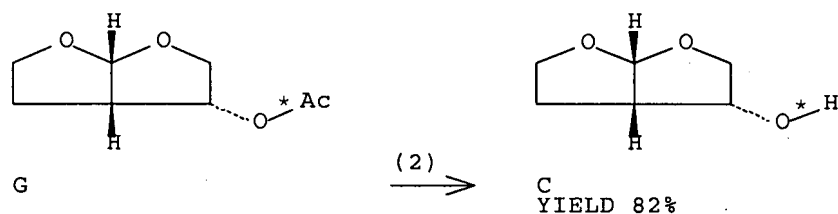
- AB Racemic bis-tetrahydrofuran ligand, (±)-hexahydrofuro[2,3-b]furan-3-ol (I), was efficiently synthesized utilizing a cobaloxime-mediated radical cyclization as the key step. I was prepared as intermediate for [3-[3-[[[(1,1-dimethylethyl)amino]carbonyl]octahydro-2(1H)-isoquinolinyl]-2-hydroxy-1-(phenylmethyl)propyl]carbamate hexahydrofuro[2,3-b]furan-3-yl ester II. Optical resolution of the racemic alc. with immobilized-Amano lipase, afforded optically pure ligands, i.e., [3R-(3α,3aβ,6aβ)]-hexahydrofuro[2,3-b]furan-3-ol and [3S-(3α,3aβ,6aβ)]-hexahydrofuro[2,3-b]furan-3-ol.
- ST HIV protease inhibitor ligand resoln; isoquinolinylhydroxypropyl carbamate furofuranyl prepn intermediate
- IT 23295-32-1  
 RL: CAT (Catalyst use); USES (Uses)  
 (preparation of hexahydrofuro[2,3-b]furan-3-yl  
 [[(aminocarbonyl)isoquinoliny  
 1]hydroxypropyl]carbamate)
- IT 1191-99-7, 2,3-Dihydrofuran  
 RL: RCT (Reactant); RACT (Reactant or reagent)  
 (preparation of hexahydrofuro[2,3-b]furan-3-yl  
 [[(aminocarbonyl)isoquinoliny  
 1]hydroxypropyl]carbamate)
- IT 109789-17-5P 156928-09-5P, [3R-(3α,3aβ,6aβ)]-Hexahydrofuro[2,3-b]furan-3-ol 156928-10-8P, [3S-(3α,3aβ,6aβ)]-Hexahydrofuro[2,3-b]furan-3-ol 162020-29-3P, [3S-(3α,3aβ,6aβ)]-Hexahydrofuro[2,3-b]furan-3-ol acetate 162119-33-7P 162119-35-9P 180902-29-8P 186488-43-7P  
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
 (preparation of hexahydrofuro[2,3-b]furan-3-yl  
 [[(aminocarbonyl)isoquinoliny  
 1]hydroxypropyl]carbamate)
- IT 107-19-7P, Propargyl alcohol 156879-13-9P, L-739,594  
 RL: SPN (Synthetic preparation); PREP (Preparation)  
 (preparation of hexahydrofuro[2,3-b]furan-3-yl  
 [[(aminocarbonyl)isoquinoliny  
 1]hydroxypropyl]carbamate)





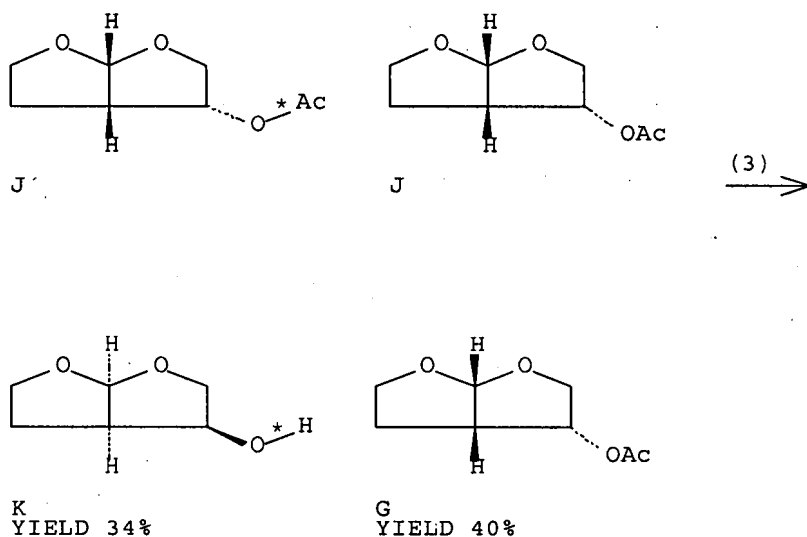
RX(1)     RCT   A 162119-33-7, B 108-24-7  
            PRO   C 156928-09-5, D 162020-29-3  
            CAT   9001-62-1 Lipase  
            SOL   110-71-4 (CH<sub>2</sub>OMe)<sub>2</sub>  
            CON   3 hours, 23 deg C  
            NTE   stereoselective, solid-supported catalyst, enzymic,  
                  biotransformation, immobilized lipase PS30 used, ee 95% for  
                  alcohol

RX(2) OF 25     ...G ==> C



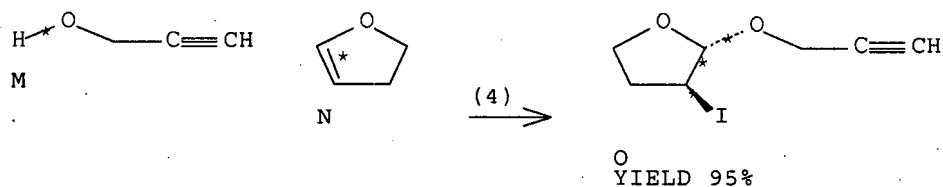
RX(2)     RCT   G 162119-35-9  
            RGT   H 917-54-4 MeLi  
            PRO   C 156928-09-5  
            SOL   109-99-9 THF  
            CON   SUBSTAGE(1) 0 deg C  
                  SUBSTAGE(2) 3 hours, 0 deg C -> 23 deg C  
            NTE   ee 94%

RX(3) OF 25     ...2 J ==> K + G...



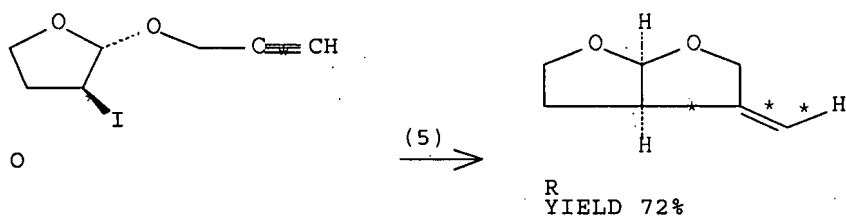
RX(3)      RCT   J 186488-43-7  
             PRO   K 156928-10-8, G 162119-35-9  
             CAT   9001-62-1 Lipase  
             SOL   7732-18-5 Water  
             CON   24 hours, 23 deg C, pH 7  
             NTE   stereoselective, solid-supported catalyst, enzymic,  
                  biotransformation, phosphate buffered solution used, immobilized  
                  lipase PS30 used

RX(4) OF 25      M + N ==> O...



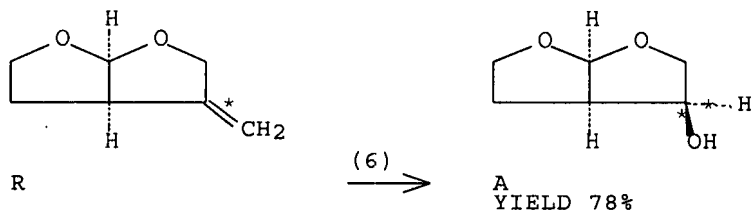
RX(4)      RCT   M 107-19-7, N 1191-99-7  
             RGT   P 516-12-1 Iodosuccinimide  
             PRO   O 180902-29-8  
             SOL   75-09-2 CH<sub>2</sub>Cl<sub>2</sub>  
             CON   SUBSTAGE(1) 0 deg C  
                  SUBSTAGE(2) 3 hours, 0 deg C -> 23 deg C  
             NTE   stereoselective, regioselective

RX(5) OF 25      ...O ==> R...



RX(5)     RCT   O 180902-29-8  
           RGT   S 16940-66-2 NaBH<sub>4</sub>  
           PRO   R 109789-17-5  
           CAT   23295-32-1 Cobalt, bis[[2,3-butanedione di(oximato-κN)](1-  
                   )]chloro(pyridine)-, (OC-6-42)-  
           SOL   7732-18-5 Water, 64-17-5 EtOH  
           CON   3 hours, 65 deg C  
           NTE   stereoselective, key step

RX(6) OF 25     ...R ==> A...



RX(6)     RCT   R 109789-17-5

STAGE(1)

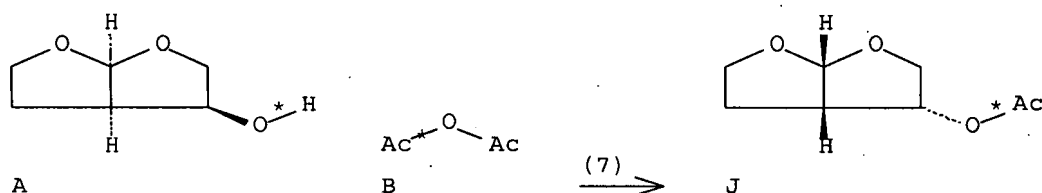
RGT   V 75-18-3 Me<sub>2</sub>S, W 10028-15-6 Ozone  
 SOL   67-56-1 MeOH, 75-09-2 CH<sub>2</sub>Cl<sub>2</sub>  
 CON   SUBSTAGE(1) -78 deg C  
       SUBSTAGE(2) -78 deg C -> 23 deg C

STAGE(2)

RGT   S 16940-66-2 NaBH<sub>4</sub>  
 SOL   64-17-5 EtOH  
 CON   1 hour, -15 deg C

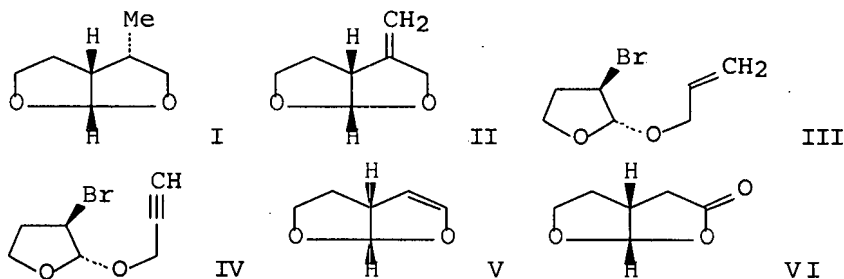
PRO   A 162119-33-7  
 NTE   stereoselective

RX(7) OF 25     ...A + B ==> J...



RX(7) RCT A 162119-33-7, B 108-24-7  
 RGT Y 121-44-8 Et3N  
 PRO J 186488-43-7  
 SOL 75-09-2 CH2Cl2

L11 ANSWER 4 OF 4 CASREACT COPYRIGHT 2007 ACS on STN  
 AN 107:77655 CASREACT. Full-text  
 TI A new route to perhydro- and tetrahydrofuro[2,3-b]furans via radical cyclization  
 AU Pezechk, M.; Brunetiere, A. P.; Lallemand, J. Y.  
 CS Lab. Synthese Org., Ec. Polytech., Palaiseau, Fr.  
 SO Tetrahedron Letters (1986), 27(32), 3715-18  
 CODEN: TELEAY; ISSN: 0040-4039  
 DT Journal  
 LA English  
 CC 28-2 (Heterocyclic Compounds (More Than One Hetero Atom))  
 GI



AB Perhydrofuro[2,3-b]furans I and II were prepared in almost quant. yields by the radical cyclization of unsatd. bromo acetals III and IV, resp., in the presence of Bu<sub>3</sub>SuH. II was transformed into tetrahydro derivative V in 4 steps. The radical annulation of ICH<sub>2</sub>CO<sub>2</sub>SnBu<sub>3</sub> to 2,3-dihydrofuran gave perhydro[2,3-b]furanone VI.  
 ST furofuran perhydro tetrahydro; radical cyclization propynyloxybromofuran allyloxybromofuran; furan bromo allyloxy radical cyclization  
 IT Ring closure and formation  
 (homolytic, of (allyloxy)bromofuran and (propargyloxy)bromofuran, perhydrofurofurans from)  
 IT 1191-99-7, 2,3-Dihydrofuran  
 RL: RCT (Reactant); RACT (Reactant or reagent)  
 (bromination of)  
 IT 109789-20-0P  
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(preparation and elimination reaction of)

IT 109789-18-6P  
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
 (preparation and hydride reduction of)

IT 109789-17-5P  
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
 (preparation and ozonolysis of)

IT 109789-14-2P 109789-15-3P  
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
 (preparation and radical cyclization of)

IT 20245-14-1P  
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
 (preparation and reaction of, with allyl alc. and propargyl alc.)

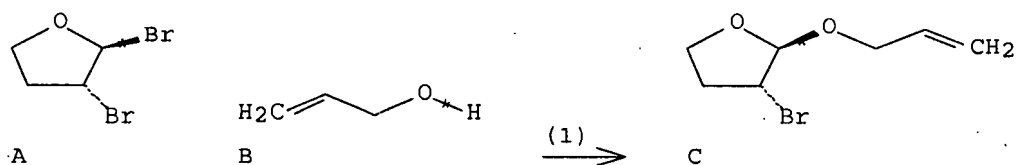
IT 109789-19-7P  
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
 (preparation and tosylation of)

IT 88938-72-1P 109789-16-4P 109789-21-1P  
 RL: SPN (Synthetic preparation); PREP (Preparation)  
 (preparation of)

IT 73927-91-0  
 RL: RCT (Reactant); RACT (Reactant or reagent)  
 (radical annulation reaction of, with dihydrofuran)

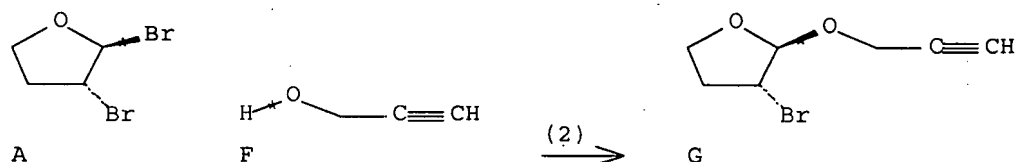
IT 107-18-6, reactions 107-19-7  
 RL: RCT (Reactant); RACT (Reactant or reagent)  
 (reaction of, with bromofuran)

RX(1) OF 34 ...A + B ==> C...



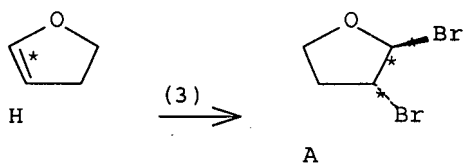
RX(1) RCT A 20245-14-1, B 107-18-6  
 RGT D 1122-58-3 4-DMAP  
 PRO C 109789-14-2  
 SOL 75-09-2  $\text{CH}_2\text{Cl}_2$

RX(2) OF 34 ...A + F ==> G...



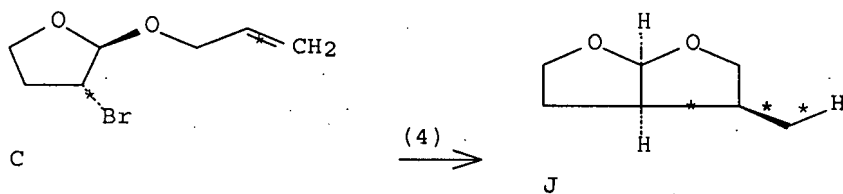
RX(2) RCT A 20245-14-1, F 107-19-7  
 RGT D 1122-58-3 4-DMAP  
 PRO G 109789-15-3  
 SOL 75-09-2 CH<sub>2</sub>Cl<sub>2</sub>

RX(3) OF 34 H ==> A...



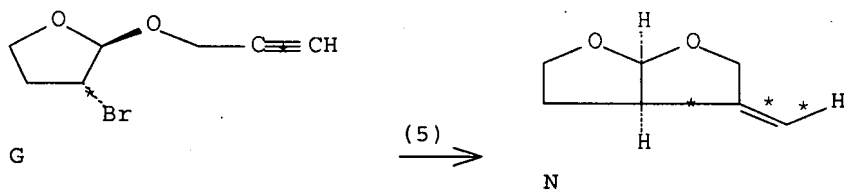
RX(3) RCT H 1191-99-7  
 RGT I 7726-95-6 Br<sub>2</sub>  
 PRO A 20245-14-1  
 SOL 75-09-2 CH<sub>2</sub>Cl<sub>2</sub>

RX(4) OF 34 ...C ==> J



RX(4) RCT C 109789-14-2  
 RGT K 688-73-3 Bu<sub>3</sub>SnH, L 78-67-1 AIBN  
 PRO J 109789-16-4  
 SOL 71-43-2 Benzene

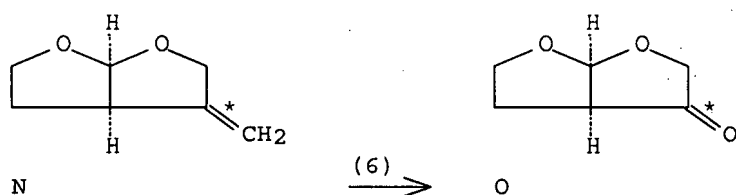
RX(5) OF 34 ...G ==> N...



RX(5) RCT G 109789-15-3

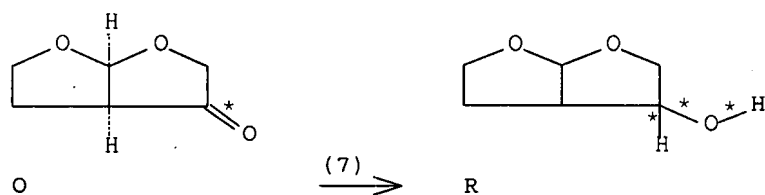
RGT K 688-73-3 Bu<sub>3</sub>SnH, L 78-67-1 AIBN  
 PRO N 109789-17-5  
 SOL 71-43-2 Benzene

RX(6) OF 34 ...N ==> O...



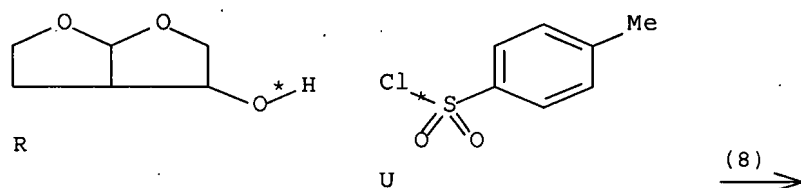
RX(6) RCT N 109789-17-5  
 RGT P 10028-15-6 Ozone, Q 75-18-3 Me<sub>2</sub>S  
 PRO O 109789-18-6  
 SOL 75-09-2 CH<sub>2</sub>Cl<sub>2</sub>

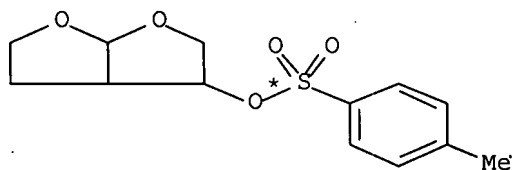
RX(7) OF 34 ...O ==> R...



RX(7) RCT O 109789-18-6  
 RGT S 16853-85-3 LiAlH<sub>4</sub>  
 PRO R 109789-19-7  
 SOL 60-29-7 Et<sub>2</sub>O

RX(8) OF 34 ...R + U ==> V...

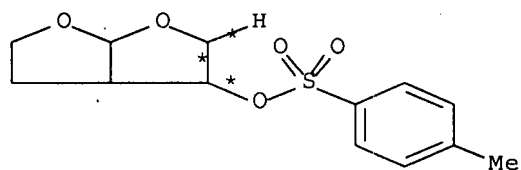




V

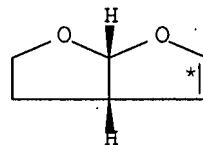
RX(8) RCT R 109789-19-7, U 98-59-9  
 PRO V 109789-20-0  
 SOL 110-86-1 Pyridine

RX(9) OF 34 ...V ==> X



V

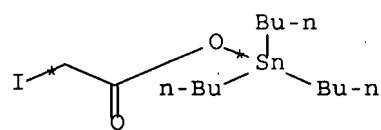
(9) →



X

RX(9) RCT V 109789-20-0  
 RGT Y 6674-22-2 DBU  
 PRO X 109789-21-1

RX(10) OF 34 Z + H ==> AA

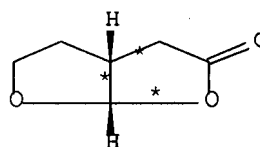


Z



H

(10) →



AA

RX(10) RCT Z 73927-91-0, H 1191-99-7  
 RGT L 78-67-1 AIBN  
 PRO AA 88938-72-1

=> d his

(FILE 'HOME' ENTERED AT 20:46:24 ON 17 AUG 2007)

FILE 'REGISTRY' ENTERED AT 20:46:33 ON 17 AUG 2007

L1 STRUCTURE UPLOADED

L2 0 S SSS SAM L1



L3 28 S SSS FULL L1  
 L4 STRUCTURE UPLOADED  
 L5 0 S SSS L4 SAM  
 L6 1 S SSS L4 FULL  
 SAVE TEMP DOAN10517966/A L3  
 SAVE TEMP DOA10517966/A L6  
 L7 29 S L3 OR L6  
  
 FILE 'CAPLUS' ENTERED AT 20:57:06 ON 17 AUG 2007  
 L8 67 S L7  
 L9 23 S L8 AND (AY<2002 OR PY<2002 OR PRY<2002)

FILE 'CASREACT' ENTERED AT 21:00:07 ON 17 AUG 2007  
 L10 21 S L7  
 L11 4 S L9 AND L10

=> save temp do10517966/a l10  
 ANSWER SET L10 HAS BEEN SAVED AS 'DO10517966/A'

=> save temp dl0517966/a l10  
 ANSWER SET L10 HAS BEEN SAVED AS 'D10517966/A'

=> fil caplus		
COST IN U.S. DOLLARS	SINCE FILE	TOTAL
	ENTRY	SESSION
FULL ESTIMATED COST	100.64	460.63
DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)	SINCE FILE	TOTAL
	ENTRY	SESSION
CA SUBSCRIBER PRICE	-2.92	-2.92

FILE 'CAPLUS' ENTERED AT 21:08:19 ON 17 AUG 2007  
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FILE COVERS 1907 - 17 Aug 2007 VOL 147 ISS 9  
 FILE LAST UPDATED: 16 Aug 2007 (20070816/ED)

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=> save temp d0517966/a l8  
 ANSWER SET L8 HAS BEEN SAVED AS 'D0517966/A'

=> save temp d517966/a l9  
 ANSWER SET L9 HAS BEEN SAVED AS 'D517966/A'

REGISTRY includes numerically searchable data for experimental and predicted properties as well as tags indicating availability of experimental property data in the original document. For information on property searching in REGISTRY, refer to:

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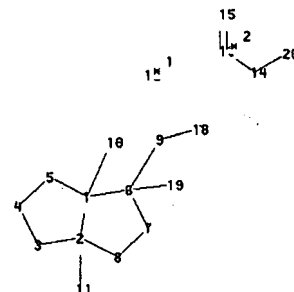
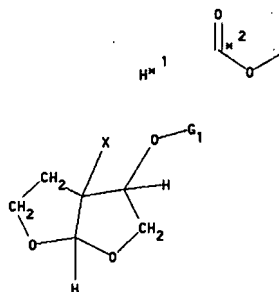
=> activate doan10517966/a

L1 STR

L2 28 SEA FILE=REGISTRY SSS FUL L1

=>

Uploading C:\Program Files\Stnexp\Queries\10517966C.str



chain nodes :

9 10 11 12 13 14 15 18 19

ring nodes :

1 2 3 4 5 6 7 8

ring/chain nodes :

20

chain bonds :

1-10 2-11 6-9 6-19 9-18 13-15 13-14 14-20

ring bonds :

1-2 1-5 1-6 2-3 2-8 3-4 4-5 6-7 7-8

exact/norm bonds :

1-2 1-5 1-6 2-3 2-8 3-4 4-5 6-7 6-9 7-8 9-18 13-15 13-14 14-20

exact bonds :

1-10 2-11 6-19

G1: [\*1], [\*2]

Match level :

1:Atom 2:Atom 3:Atom 4:Atom 5:Atom 6:Atom 7:Atom 8:Atom 9:CLASS 10:CLASS  
11:CLASS 12:CLASS 13:CLASS 14:CLASS 15:CLASS 18:CLASS 19:CLASS 20:CLASS

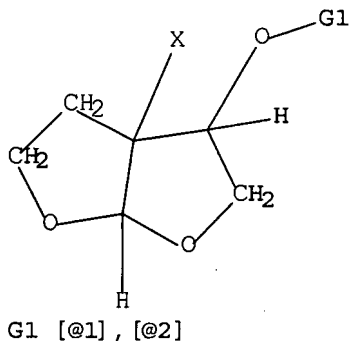
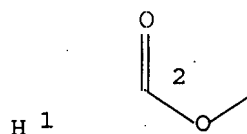
L3 STRUCTURE UPLOADED

=> d 13

L3 HAS NO ANSWERS

L3

STR



Structure attributes must be viewed using STN Express query preparation.

=> s l3 subset=l2 sam

SAMPLE SUBSET SEARCH INITIATED 21:25:47 FILE 'REGISTRY'

SAMPLE SUBSET SCREEN SEARCH COMPLETED - 0 TO ITERATE

100.0% PROCESSED 0 ITERATIONS 0 ANSWERS  
SEARCH TIME: 00.00.01

PROJECTIONS (WITHIN SPECIFIED SUBSET):	ONLINE	**COMPLETE**	
PROJECTED ITERATIONS (WITHIN SPECIFIED SUBSET):	0 TO		0
PROJECTED ANSWERS (WITHIN SPECIFIED SUBSET):	0 TO		0

L4 0 SEA SUB=L2 SSS SAM L3

=> s l3 subset=l2 full

FULL SUBSET SEARCH INITIATED 21:26:00 FILE 'REGISTRY'

FULL SUBSET SCREEN SEARCH COMPLETED - 3 TO ITERATE

100.0% PROCESSED 3 ITERATIONS 3 ANSWERS  
SEARCH TIME: 00.00.01

L5 3 SEA SUB=L2 SSS FUL L3

=> fil caplus

COST IN U.S. DOLLARS	SINCE FILE	TOTAL
	ENTRY	SESSION
FULL ESTIMATED COST	42.00	42.21

FILE 'CAPLUS' ENTERED AT 21:26:23 ON 17 AUG 2007

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FILE LAST UPDATED: 16 Aug 2007 (20070816/ED)

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=> s l5

L6 2 L5

=> fil reg

COST IN U.S. DOLLARS

SINCE FILE	TOTAL
ENTRY	SESSION
0.47	42.68

FULL ESTIMATED COST

FILE 'REGISTRY' ENTERED AT 21:27:06 ON 17 AUG 2007  
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Property values tagged with IC are from the ZIC/VINITI data file provided by InfoChem.

STRUCTURE FILE UPDATES: 16 AUG 2007 HIGHEST RN 944884-94-0  
DICTIONARY FILE UPDATES: 16 AUG 2007 HIGHEST RN 944884-94-0

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<http://www.cas.org/support/stngen/stndoc/properties.html>

=> activate doal0517966/a

L7 STR

L8 1 SEA FILE=REGISTRY SSS FUL L7

=> fil capl

COST IN U.S. DOLLARS

SINCE FILE	TOTAL
ENTRY	SESSION
0.45	43.13

FULL ESTIMATED COST